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The Effect Of Gas House Waste
On The Biochemical Oxidation Of Sewage

THE EFFECT OF GAS HOUSE WASTE ON THE BIOCHEMICAL OXIDATION OF SEWAGE

BY

CHESTER WILLIAM LENZING

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Chester William Lenzing

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IS APPROVED BY ME AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE

DEGREE OF Bachelor of Science in Chemistry

Edward Bartow

Instructor in Charge

APPROVED: W. A. Noyes

HEAD OF DEPARTMENT OF Chemistry

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THE EFFECT OF GAS HOUSE WASTE ON THE BIOCHEMICAL OXIDATION OF SEWAGE.

Introduction

The waste liquor coming from a gas house probably affords greater difficulties in purification by bacterial means than any other trade waste. This waste is supposed to be more or less antiseptic in its action. Phenols, thiocyanates and other compounds poisonous to bacterial life are usually present in the waste. They are supposed to inhibit bacterial growth and thus hinder or prevent nitrification in a stream or the production of a stable effluent from a sewage treatment plant.

It is difficult to find chemical means to measure the effect of gas house waste on the biochemical oxidation of sewage. The test for oxygen consumed would not be a true measure of the biochemical oxygen demand because the gas waste would naturally raise the oxygen consumption figure. According to Lederer[6] the oxygen consumed figure has no relation to the true biochemical oxygen demand. Dilution with methylene blue is not practical because the color of the

waste conceals the color of the methylene blue. The same determination of dissolved oxygen at the end of the incubation is also not accurate on account of the loss of oxygen. The only method for determining the effect of the waste on the biochemical oxidation of sewage is the determination of the biochemical oxygen demand by the saltpeter method as developed in the laboratory of the Sanitary District of Chicago.[7]

In this thesis there is described an attempt, by the use of the saltpeter method, to get a relation between the oxygen demand of the sewage containing no gas waste and sewage containing varying amounts of the waste.

The sewage used in this work was taken from the main Champaign sewer at the experimental activated sludge plant in the power house of the University of Illinois. The entire flow of the city of Champaign amounting to about one million gallons per day passes this point on its way to the outfall in the Salt Fork in Urbana.

The samples of liquor were taken from the Champaign Gas Works at Fifth and Hill streets, where both coal and water gas are made. Water gas is the principal product and furnishes the greater part of the waste liquor. It is difficult to accurately determine the amount of waste from the six different sources at the plant. The waste liquor used in the experimental work comes from the condensors and the scrubber and comprises the largest volume of the waste. It flows into

a settling tank, consisting of an old relief holder, where the tar is allowed to settle out. The effluent containing the lighter oils, which, up to a few months ago was emptied into the Boneyard is now run back into the condensors and is not allowed to enter the stream. The total objectionable waste is estimated at two hundred and fifty gallons per hour. This would make six thousand gallons per day. If it were emptied into the sewer it would be six tenths per cent of the Champaign sewage estimated to be one million gallons per day.[5]

Historical.

Practically all of the investigation of the effect of the contamination of sewage by gas waste has been done in England though the first recorded work was in Germany.

In 1877, H. Vohl reported a case in Creuznach, Germany of contamination of a well from a nearby gas works. The proprieter of the well brought suit against the company, but lost because the chemists were unable to detect in the water compounds known to exist in the gas liquor. Vohl showed, however, that these compounds were changed in filtration through the soil and demonstrated that the water was contaminated by large quantities of ammonia, magnesia, nitrites, and thiosulphates.[14]

In 1907, Frankland and Silvester,[4] studying the purification of a sewage containing as much as nine per cent of gas liquor in Oldbury, England, found that triple contact filters effected satisfactory purification. They found also that the vitality of B coli was considerably reduced by one hundred parts per ten thousand thiocyanate in gas liquor.

In 1910 Ardern and Lockett[1], working with gas liquor only, found that they could successfully oxidize solutions of phenol and thiocyanates on bacterial filters. They found that denitrification does not play an important role in the oxidation of thiocyanates. Phenol was more readily oxidized than the thiocyanates.

Fowler[3] in 1910 pointed out that sewage without gas liquor was oxidized weeks before that containing a samll per cent of crude gas liquor. He isolated an organism from a bacterial filter which actually oxidized weak solutions of phenol.

In 1912 Ardern and Lockett[2] continuing their work with gas liquor, studying its effect on the oxidation of sewage, showed that one per cent of Manchester gas waste had a retarding effect on the oxidation of sewage by aeration, but noticed no effect on fine grain sand filters. The permanganate absorption figure remains higher as the filter matures, but the sewage is oxidized.

The purification of gas house waste was investigated at the Massachusetts Experiment Station at Lawrence[11]. It was found that treatment with chemicals such as lime, copperas, etc., and filtration through coke was a suitable treatment. No mention is made of the effect of the waste on the oxidation of sewage.

Experimental.

Champaign sewage varies in composition from day to day and even from hour to hour. It is therefore difficult to show its average composition. The sewage is strongest at nine in the morning, at which time most of the samples used were collected. An analysis of a nine o'clock sewage made September 29, 1915 is typical.

Table I.

Analysis of a nine o'clock sewage collected September 29, 1915

	pts. per million
Total residue on evaporation	2145
Oxygen consumed	94
Ammonia Nitrogen	40
Albuminoid nitrogen	24
Total organic nitrogen	24
Nitrite nitrogen	0.
Nitrate nitrogen	1.6
Alkalinity (as $CaCO_3$) methyl orange	608.
Alkalinity (as $CaCO_3$) phenolphthalein.	48.
Chlorine	142.

The composition of the waste liquor obtained October 21, 1915, is shown in table II.

Table II.

Analysis of the crude unsettled gas liquor.

	pts. per million.
Residue on evaporation	2006
Suspended matter	1466
Alkalinity (as $CaCO_3$) methyl orange	468
Total organic nitrogen	43.4
Nitrite nitrogen	0
Nitrate nitrogen	0

Table III.

Analysis of the settled waste.

	pts. per million
Residue on evaporation	780
Suspended matter	258
Alkalinity	464
Total organic nitrogen	64
Nitrite nitrogen	0
Nitrate nitrogen	0

The sewage and gas liquor have been analyzed according to Standard Methods of Water Analysis of the American Public Health Association.

Having obtained by analysis some idea of the composition

of both sewage and gas liquor, a study was made of the effect of the liquor on the biochemical oxygen demand determined by the saltpeter method. A definite quantity of saltpeter is added to sewage, and after incubating ten days at twenty degrees Centigrade the residual nitrate and nitrite are determined. The oxygen demand is the difference between the amount of oxygen present at the beginning and the end of the period. In the test the sodium nitrate furnishes the available oxygen. Under natural conditions in a stream[8] the oxygen may come from the dissolved oxygen of the stream, may be absorbed from the atmosphere or may be furnished by the plankton. In calculating the oxygen demand by the saltpeter method the following assumptions were made[9]:-

(1) That every molecule of sodium nitrate furnishes 2 1/2 atoms of oxygen.

(2) That every molecule of sodium nitrite furnishes 1 1/2 atoms of oxygen.

Calculating this relation using nitrate nitrogen and nitrite nitrogen instead of sodium nitrate and sodium nitrite, it can be seen that one part of nitrate nitrogen by weight is equivalent to 2.85 parts by weight of oxygen. Likewise one part of nitrite nitrogen will represent 1.7 parts available oxygen by weight.

There might be some objection to the use of the sodium nitrate method on sewage containing a waste which is anti-

septic in its character, on the ground that in the 250 cc. bottles used in the laboratory experiment, the disinfectant is more concentrated than it would be in actual treatment. Since the efficiency of a disinfectant depends greatly upon its strength in a solution, this is perhaps a serious objection. In treatment of sewage by dilution with fresh water the disinfecting action would be weakened, but in aerobic treatment without dilution the conditions of the experiment would be more like the actual conditions of treatment. It must be remembered also, that laboratory methods do not usually work out as well in practice.

Lederer[8] recommends that the waste should be slightly alkaline and never acid. The analysis of this waste at the beginning of the work shows it to be alkaline and not acid. (See tables 2 and 3).

Biochemical oxygen demand by the Sodium Nitrate Method[6-7-9-10]

Reagents:- Sodium nitrate solution. 26.56 grams C P. sodium nitrate per liter. 1 cc of this solution in 250 cc sewage represents 50 parts per million available oxygen.

Proceedure:- The saltpeter solution was added to the sewage and gas waste in bottles holding approximately 250 cc which were completely filled and stoppered. The bottles which varied more than eight cubic centimeters were rejected. The sodium nitrate solution was added from a burette. The sewage was added from an aspirator bottle fitted with a spiral stirrer to keep the solid particles of the sewage evenly distributed. This stirrer consisted of a stiff wire made into a spiral at one end.

The spiral was never allowed to come in contact with the air and cause artificial aeration. The same care was exercised in filling the bottles with sewage. The glass tube of the aspirator bottle was inserted to the bottom of the bottle before allowing the sewage to enter, and was drawn up as the level of the liquid rose. Thus aeration was avoided. The temperature was kept as near to that of incubation as possible. The samples were then incubated at 20° for 10 days. Each day a bottle was taken from the incubator and the oxygen demand was calculated from determinations of nitrate and nitrite nitrogen.

Nitrate nitrogen was determined by the aluminium reduction method outlined in Standard Methods of Water Analysis followed by direct Nesslerization. Nitrite nitrogen was determined by the sulphanillic acid-napthylamine method. In most cases the determinations were made immediately, but if a delay was necessary the samples were preserved with chloroform on ice until the following day.

The first series is disregarded. Owing to lack of familiarity with the method the results are inaccurate and therefore conclusions can not be drawn from them.

In the second series five sets of fifteen bottles each were incubated. Sets 1, 2, 3 and 4 contained 400, 450, 500 and 500 parts per million available oxygen, respectively in each bottle and no gas liquor. Set 5 contained 500 parts per

million oxygen and 10 per cent crude gas liquor.

The oxygen demand of the five sets in the second series differ from each other. (See table 4 and Fig. 1.). The difference may be due to variation in the concentration of oxygen. In each set after the first, the available oxygen is increased by 50 parts per million, and according to the Law of Mass Action the speed of the reaction varies with the concentration. Lederer[8,9] claims that better results are obtained by keeping the available oxygen near that of the oxygen demand and our later work verifies this claim. The oxygen demand on the whole varied directly with the time in the sets in series 2. (See Fig.1). Several individual determinations did not conform to the general rule. This is probably due to error in the determination of such large quantities of residual nitrate and nitrite by colorimetric methods. Lederer[6] found discrepancies in most of his series, which he was unable to explain. He claimed that the error in results should never exceed 10 per cent even in the results obtained by the inexperienced chemist. In our experience only with care can the error be reduced to 10 per cent. To illustrate, if in a determination 2 cc of sodium nitrate solution is added to 250 cc of sterile water, the available oxygen is 100 parts per million. When the nitrates are determined, 5 cc of the solution is diluted to 50 cc and (after evaporation to 15 cc, transference to a Nessler tube, dilution again to 50 cc, the addition of aluminium and the reduction over night), 10 cc of

this solution is taken. This is the largest quantity that can be used for comparison with Nessler standards. Assuming that reduction was complete and that the measurements with pipettes were accurate, the only error would be in reading the color. The closest one can read is to 0.2 cc of the standard. When 100 parts per million available oxygen is present, the difference of 0.2 cc would make a difference of 2 parts per million nitrogen, which when multiplied by 2.85 will give an error of 6 parts per million or 6 per cent of the 100 parts per million of oxygen.

The color reaction for nitrites is far more sensitive and is likely to lead to greater errors because of the higher dilution necessary. In the sewage alone the nitrites remain less than 20 parts per million, while in sewage containing gas waste the nitrites may increase to 100 parts per million.

Our conclusion from the second series by comparison of sets 2, 3, 4, with set 5 of series 2, is that the presence of 10 per cent gas waste in sewage has no greater effect on the biochemical oxygen demand than the possible error in analysis. With care the experimental error can be reduced to 10 per cent but with the large quantities of residual nitrate and nitrite nitrogen, it is likely that the error runs above 10 per cent.

In order to eliminate the possible source of error due to excessive residual nitrate, less sodium nitrate, containing the equivalent of only 150 parts per million oxygen, was added

Table 4

Comparison of the Oxygen Demand of Sewage with and without gas liquor. Series 2.

Days Incubation	400 ppm O Set 1	450 ppm O Set 2	500 ppm O Set 3.	10 % gas waste 500 ppm O Set 4	Set 5
.5	186	238	263	258	218
1.0	186	241	261	227	216
1.5	195	245	190	187	188
2.0	195	245	281	189	188
2.5	195	245	124	129	55
3.0	138	245	238	248	159
3.5	229	257	227	237	113
4.0	125	177	227	233	205
4.5	125	253	404	304	242
5.0	273	344	349	326	244
6.0	273	322	372	340	250
7.0	296	348	341	340	330
8.0	325	371	328	310	250
9.0	267	281	304	286	233
10.0	278	253	269	304	lost.

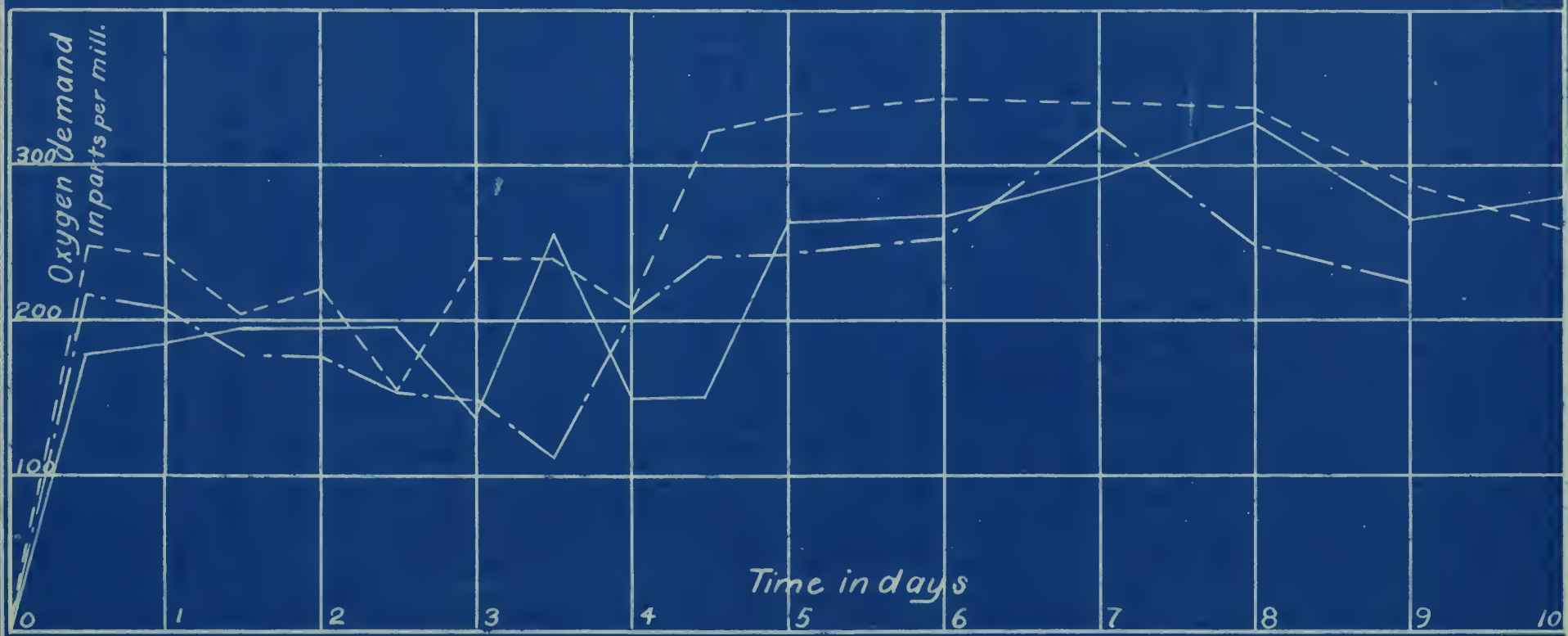

Figure 1

Graph showing relation between Oxygen Demand and time Series 2

Set 1 – 400 p.p.m. available oxygen ————

Average Sets 2, 3 & 4 450 - 500 p.p.m oxygen – – – – –

Set 5 500 p.p.m O. 10% gas waste —— · ——

to the bottles to be incubated for 1 day. The amount of oxygen was increased by 50 parts per million per day for the four succeeding days. The results obtained were more satisfactory but the gas liquor still showed practically no interference. (See Table 5 and Fig. 2).

Table 5

Comparison of the Oxygen Demand of Sewage with and without gas liquor. Series 3.

Days Incubation	Available O added.	Residual O from NO_2		Residual O from NO_3		Oxygen demand	
	Parts per million						
		Sets* 1 & 2	Sets** 3 & 4	Sets* 1 & 2	Sets** 3 & 4	Sets* 1 & 2	Sets** 3 & 4
1	150	17	1.8	67	110	66	25
2	200	32	93	73	0	105	106
3	250	32	127	105	0	113	120
4	250	25	144	79	0	140	110
5	300	34	153	136	0	130	117
6	350	23	114	204	96	132	140
7	350	14	116	206	104	130	151
8	350	16	93	183	147	150	132
9	350	20	122	174	112	152	125
10	350	15	170	182	34	157	146

*No gas liquor

** 10% gas liquor.

The oxygen demand of sewage containing 10 per cent gas liquor is for the most part below that of the sewage containing no gas waste but not enough lower to warrant any conclusion that the liquor had affected the oxygen demand. In the sewage alone the nitrite is low while in the sewage to which gas liquor was added it is high. The oxygen demand must therefore be calculated from two different sources. The error in the determination may be enough to make the difference in figures obtained.

The difference in residual nitrite nitrogen was very marked see table 6, and is difficult to explain. The gas liquor may slow up the reaction in such a way that the nitrate is reduced only to the nitrite. It may be that the gas liquor inhibits the action of some of the bacterial forms which denitrify from nitrite to ammonia or free nitrogen.

TABLE 6

Residual Nitrite Nitrogen from Series 3.

Days incubation	no gas waste	10 % waste
1	10	1
2	18	55
3	19	75
4	15	85
5	20	90
6	13	68
7	8	70
8	10	55
9	12	75
10	9	100

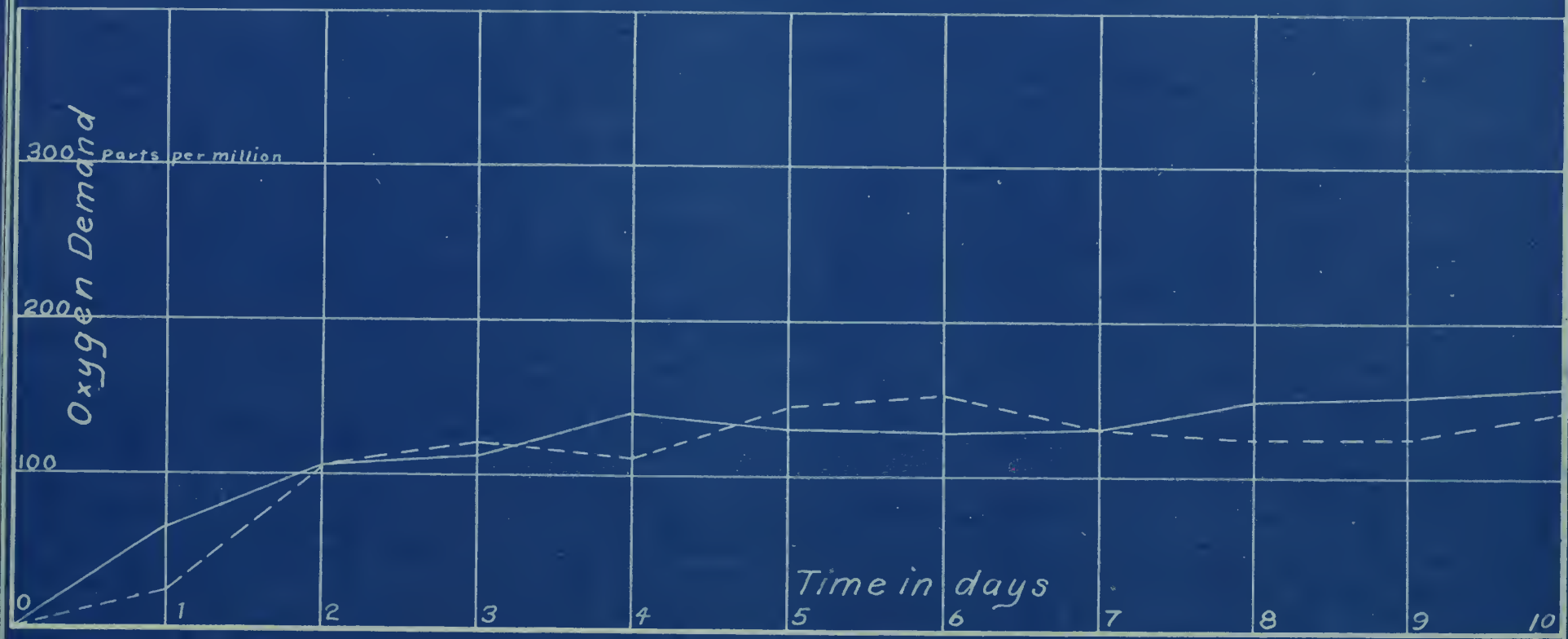

Figure 2

Graph showing relation between Oxygen Demand and time. Series 3

Average of Sets 1 & 2 ———

Average of Sets 3 & 4 - - - -

Another series in which 15 per cent gas liquor instead of 10 per cent was used, showed no interference by the gas liquor.

Effect of Gas Liquor on Sodium Nitrate in the Absence of Bacteria.

The gas liquor has no effect on sodium nitrate in the absence of bacteria, for when 15 per cent waste in sterile water was incubated for 8 days with sodium nitrate equivalent to 100 parts per million available oxygen, there was practically no action on the nitrate. (See Table 7).

TABLE 7

Action of Gas House Waste on Sodium Nitrate in the absence of Bacteria.

Days Incubation	Nitrite as N parts per million	Free NH_3	Nitrate
0	0	8.8	38
8	0.5	7.2	36

Action of Gas House Liquor on Bacteria

The liquor itself was sterile, for when 1 cc of the liquor, undiluted, was plated in the usual way on agar no growth was found on the plate after an incubation period of 24 hours at 37 degrees Centigrade.

350 parts per million oxygen in sodium nitrate was added to each of 4 bottles of sewage containing 0.5 per cent, 10 per cent, 15 per cent of gas liquor, which were then incubated at 20 degrees Centigrade. The number of bacteria growing on agar incubated at 37 degrees Centigrade was determined at 24 hour intervals. Instead of the expected decrease in the number of bacteria in the sewage containing the gas liquor, an increase was observed. (See Table 8).

Table 8 .

Bacterial counts on agar made from sewage with and without gas house waste.

% gas liquor	3 hrs.	1 day.	3 days.	8 days.
0	850,000	1,700,000	400,000	200,000
5	1,500,000	1,200,000	1,200,000	no count
10	1,200,000	1,900,000	2,500,000	24,000,000
15	280,000	1,700,000	2,600,000	44,000,000

Two duplicate series also showed an increase. These results would lead to the conclusion that the gas liquor in sewage is favorable for the growth of certain bacterial flora. No special attempt was made to study the forms found on the agar plates, except that at the end of 8 days colonies were picked from the plates and inoculated in nutrient broth

to which had been added 15 per cent gas liquor. After 24 hours a good growth was obtained in all the broth tubes.

These bacteriological results show that amounts of gas waste as high as 15 per cent have no inhibiting action on the number of bacteria in sewage.

Strength of Gas Liquor.

Since gas liquor has a varying composition, and a varying concentration need was felt for some method by which waste flowing at one time could be compared with that at another. Determinations of ammonia nitrogen, thiocyanates, and phenol were chosen as suitable tests of the strength of gas liquor. On account of the ease with which the determinations could be made ammonia nitrogen and thiocyanates were determined in the samples collected every two hours during a twelve hour run at the gas works. The results were found to vary sufficiently to make a comparison with a standard necessary. (See Table 9).

Methods of Analysis

The ammonia nitrogen was determined according to Standard Methods of Water Analysis.[12]

Thiocyanate was determined colorimetrically by comparing in Nessler tubes, the unknown solution to which the same excess of ferric chloride was added, with standard solutions

Table 9

Ammonia nitrogen and Thiocyanate in Gas Liquor.

Time of collection of sample	Ammonia Nitrogen	Thiocyanate.
7 AM	1150 p.p.m.	1900 p.p.m.
9 AM	900 "	1800 "
11 AM	1000 "	2200 "
1 PM	1350 "	2300 "
3 PM	1650 "	3400 "
6 PM	1850 "	3700 "

containing varying amounts of ammonium thiocyanate, a gram sodium chloride per liter, and a definite quantity of ferric chloride. If the solution was dilute enough phenol would not interfere. A standard containing no thiocyanate was made up in order to get a zero reading. The unknown solution was compared with the standards before adding ferric chloride to eliminate any error caused by the natural color of the gas liquor.[2]

The determinations of phenol in gas liquor[13].

Reagents,- Solution of Ammonium polysulphide
Solid lead carbonate
Solid calcium carbonate
N/10 sodium hydroxide.
N/10 Iodine solution.
N/10 Thiosulphate solution.
Starch solution.

Procedure,- "100 cc of the filtered gas liquor is first treated with an excess of ammonium polysulphide to convert any cyanide present into cyanate. This is allowed to stand, and the solution made up to 200 cc. The sulphides are then precipitated, most conveniently by lead carbonate, and the lead sulphide and excess of sodium hydroxide (25 cc of 50 per cent solution) is then added to 100 cc of the filtrate, and the solution evaporated until the salts begin to crystallize out. The residue is washed into a liter distilling flask and diluted to about 150 cc with distilled water, and when cold is acidified with sulfuric acid. The liquid is distilled until the salts begin to crystallize out; we thus obtain distillate 1. 100 cc of water is added to the distilling flask, and the acid liquid again distilled into a second receiver, giving distillate 2. This is repeated a third time giving distillate 3. The whole of the phenol will usually be in these three distillates. In order to get rid of any acid which was distilled over, and which would upset the subsequent titration, each of the distillates is then shaken with about 1 gram of calcium carbonate (and a small quantity of lead carbonate) and redistilled. In doing this distillate #1 is distilled first, #2 is then added to the distilling flask and distilled, #3 being distilled last. The final distillates are then united and made up to 500 cc, when they are ready for titration.

"The titration was carried out as follows,- 100 cc of the united distillate was made alkaline with sodium hydroxide and warmed to 60 degrees Centigrade. An excess of iodine

was then run into the flask which was immediately stoppered and allowed to cool with frequent shaking. The contents were then titrated back with N/10 thiosulphate solution, starch being used as an indicator. The end point is marked by a transition from blue to a rose pink.

"The reactions involved are as follows,-

$$2\ NaOH + 2\ I \rightarrow NaIO + NaI + H_2O$$

(1) $C_6H_5OH + 3\ NaIO \rightarrow C_6H_2I_3OH + 3\ NaOH$

(2) These reactions are equivalent to,-

$$C_6H_5OH + 6\ I \rightarrow C_6H_2I_3OH + 3\ HI$$

On acidification the following reaction takes place,-

$$NaIO + NaI + H_2SO_4 \rightarrow I_2 + Na_2SO_4 + H_2O"$$

The method was tried out on a known solution of phenol containing 1.7167 gram phenol. The amount found by analysis by this method was 1.708 gram phenol, an error of 0.5 per cent. Phenol in two samples of liquor collected at different times was then determined and found to be 213 and 433 parts per million respectively.

Standardization of the waste by odor was suggested at one time, and on making dilution it was found that this method was not delicate enough.

Conclusions.

In conclusion,- the study of the effect of the gas house waste on the biochemical oxidation of sewage has shown,-

1. By use of the saltpeter method for determining the biochemical oxygen demand, with its large possible experimental errors, no effect was noticed on the biochemical oxidation of sewage by 10 and even 15 per cent of the gas waste.

2. The presence of the gas waste in sewage containing sodium nitrate has the effect of increasing the nitrite nitrogen after incubation, over that of sewage containing no gas waste.

3. That 15 per cent of the gas house waste has no inhibiting action on the number of bacteria as shown by counts made on agar incubated at 37 degrees. An increase in the number of bacteria was observed in sewage containing gas waste over sewage containing no gas waste.

4. Determinations of ammonia nitrogen and thiocyanate were found sufficient tests for comparing waste at one time against waste at another.

Finally the author wishes to acknowledge the kindly interest in the progress of this work shown by Messrs. Langelier, Mohlman, and Greenfield of the Illinois State Water Survey, and Doctor Lederer, Bacteriologist of the Sanitary District of Chicago; to thank Mr. Condon, Superintendent of the Champaign Gas Plant for his cooperation in collecting the samples of liquor; Dr. Corson for suggesting the topic, and

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